

BOILING TEMPERATURES OF KJELDAHL DIGESTION MIXTURES*

By C. L. OGG and C. O. WILLITS
(Eastern Regional Research Laboratory¹)

The widely divergent results obtained in the 1948 collaborative studies of the micro Kjeldahl procedure emphasized that inherent errors existed in the methods generally used. A report of work at this Laboratory (1)

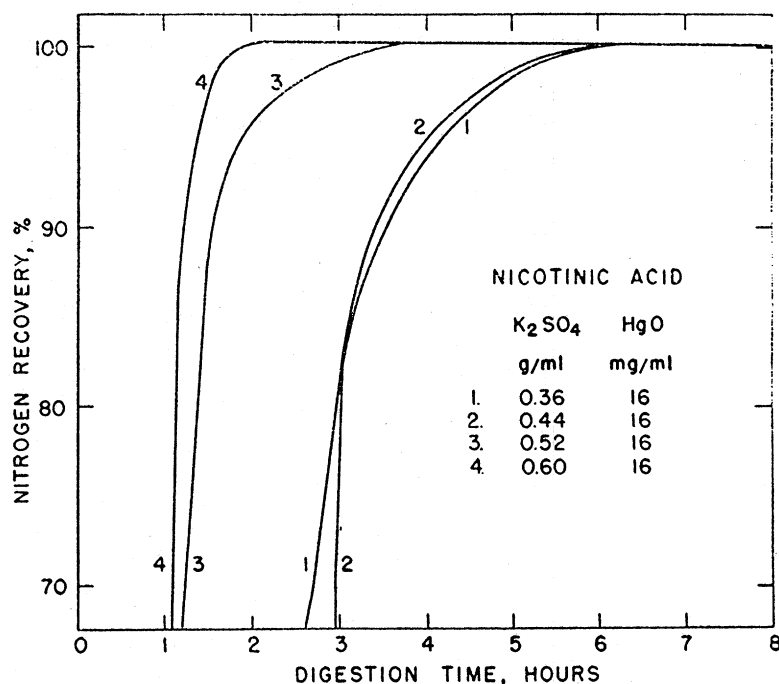


FIGURE 1

FIG. 1.—Effect of Time of Digestion and Potassium Sulfate Concentration on Recovery of Nitrogen.

indicated that not only was the salt concentration critical but also that the required time of digestion and the amount of salt were closely related (Figure 1). As a result, the 1949 collaborators were instructed to use a high

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¹ One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

were separated by a porcelain rod insulator, which also served as a handle to place the thermocouple bead in the desired location. The thermocouple was checked against a calibrated 360°C. thermometer.

Table 1 shows the temperatures at the locations indicated in Figure 2. Although the concentration of potassium sulfate ranged from 0.25 to 0.875 g. per ml. of sulfuric acid, the three concentrations of greatest interest were 0.375, 0.50, and 0.625 g./ml., since they cover the range of concentrations more commonly used. These three concentrations correspond closely to 9, 12, and 15 g. of potassium sulfate per 25 ml. of sulfuric acid.

If the temperature differences of the solutions (location 2) are compared with the digestion rate curves of nicotinic acid (Figure 1), it will be noted that the rate of digestion as shown by minimum digestion times is roughly

TABLE 1.—Boiling temperatures of Kjeldahl digestion mixtures
Temperature, °C.

Grams of K₂SO₄ per ml. H₂SO₄

LOCATION	0.250	0.375	0.500	0.625			0.750	0.875
1	334*	342*	345*	353*	356†	356‡	360*	365*
2	332	336	342	349	349	349	354	358
3	332	336	342	349	349	348	353	354
4	327	333	333	342	344	345	345	348
5	315	325	318	334	341	338	323	339
6	265	270	272	282	288	285	278	283

* Readings made in 30-ml. Kjeldahl flasks on micro gas digestion rack.

† Readings made in 500-ml. Kjeldahl flask on macro electric digestion rack.

‡ Readings made in 30-ml. Kjeldahl flask on micro electric digestion rack.

doubled for each 10°C. increase in temperature. For example, the minimum time for complete digestion for potassium sulfate concentrations of 0.60 and 0.36 g. per ml. was 2 and 6 hours, respectively, and the difference between the boiling temperatures of the two corresponding solutions, 0.625 and 0.375 g. per ml. was 13°C.

Since the reaction rate appears to be so dependent on temperature, the amount of potassium sulfate and the digestion time are critical, especially when analyzing refractory nitrogenous materials. The amount of potassium sulfate and time of digestion usually used probably exceed the critical limits for easily decomposed materials such as amides and amines. However, when the collaborators studying the micro Kjeldahl method in 1948 were asked to analyze nicotinic acid, their results (2) ranged from less than 1 to 11.5 per cent nitrogen (theoretical value, 11.38 per cent).

In the light of the data presented here, this great discrepancy of results may be attributed principally to the boiling temperatures of the different digestion mixtures or to the use of insufficient heat. In all cases, the excess water must be distilled from the flask and the remaining solution boiled

briskly. In the temperature range of the boiling points of these different mixtures, the minimum time of digestion varies inversely with the temperature, that is, the higher the boiling temperature the shorter the digestion time. Apparently the time can be further shortened by superheating the layer of digestion mixture in contact with the walls of the flask. The minimum digestion time for nicotinic acid by the macro procedure was 2 hours, whereas 3 hours were required when using the micro gas heated rack. The temperatures of the boiling mixtures were the same (Table 1, location 2, columns 4 and 5), but the temperature at the bottom of the electrically heated macro flask (location 1,) was 7°C. higher than that of the liquid (location 2); the temperature difference between these locations in the gas-heated micro flask was only 4°.

LITERATURE CITED

- (1) WILLITS, C. O., COE, M. R., and OGG, C. L., *This Journal*, **32**, 118 (1949).
- (2) WILLITS, C. O., and OGG, C. L., *Ibid.*, **31**, 565 (1948).